

Design Features of a Commercial Carbon-in-Ash Monitor
Based on the Photoacoustic Effect

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The utility industry would like to find a practical and convenient means to monitor either, on or off line, the amount of unburned carbon in coal ash. The interest is the efficient use of coal while meeting low NO_x emission requirements. In addition, the potential use of fly ash as a filler in a variety of applications dictates that the elemental carbon be monitored and controlled particularly in regard to adsorption i.e., surface area. A principle interest is on the adsorption of surfactants by carbon in fly ash, which is used as a filler in concrete. The resulting lack of air retention by the concrete is detrimental to performance in cold climates.

At last years conference a presentation was given on comparing photoacoustic, loss-on -ignition and foam index methods to determine the carbon in fly ash. Work has continued in developing an instrument, designated CA200, and the comparison has been extended to concentrating on measuring the amount of carbon in the fly ash. Strides have been made in improving signal level and rejection of ambient noise. Even though the industry is aware of the errors possible with either organic carbon LOI or plain LOI it has proven adequate to detect carbon trends. We have therefore taken the position that the user would thereby be the most familiar with readout directly associated with LOI. Our improved calibration standards and zeroing methods have been directed at increasing the correlation between photoacoustic techniques and LOI.

Ametek Corporation and Iowa State University are continuing to work together to develop photoacoustic technology as a means to easily and practically measure the mass of carbon in fly ash. By lowering the acoustic operating frequency into the subsonic region, the signal level of the CA200 photo acoustic instrument has been increased three orders of magnitude into the millivolt range. An economical visible LED diode is retained as a light source as well as a readily available commercial microphone for pressure wave detection. Synchronous monitoring is used to select and isolate the signal from the environment. A

simple sample introduction/sealing system, as well as a microprocessor user interface, has been developed to make a user-friendly instrument. A drop through grinder is used to bring the surface to volume ratio of the relatively soft carbon to a constant value. This approach offsets variations and allows the instrument readings to correspond to the mass of carbon in the sample.

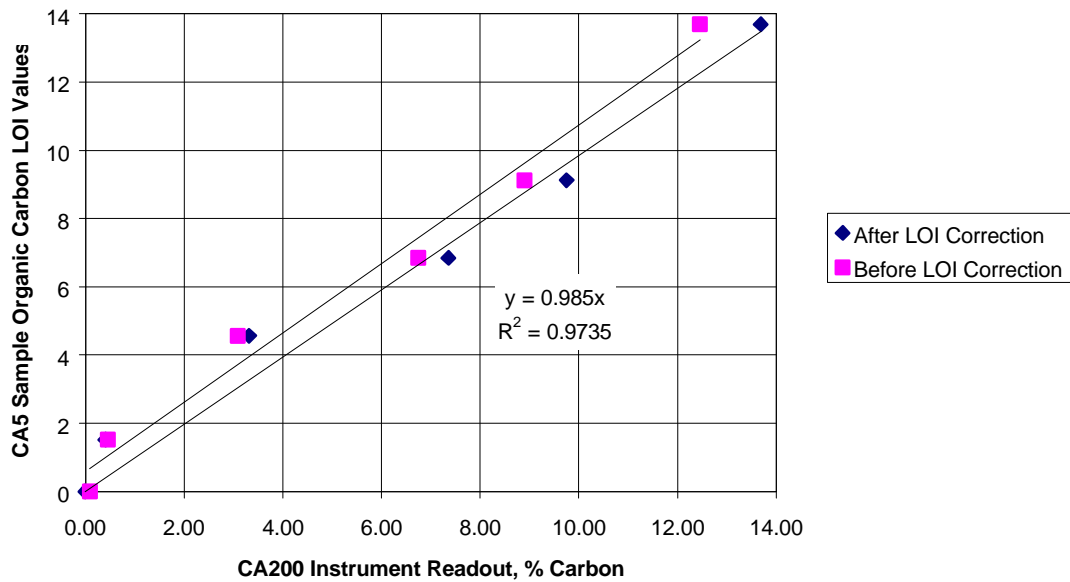
A series of 30 samples from China, Australia and the United States were examined using a ground samples and their corresponding zeroes. Zero ash is made by firing a sample in air to 750 degrees C to remove carbon. The samples have proven to be widely varied and were on site stored in the dry and wet state.

A calibration curve was developed using a sample of high carbon fly ash, which was cut by weight using zero ash obtained by firing a portion of the same ash. This approach using fly ash carbon was aimed at eliminating the error associated with the variations in the crystalline nature of the carbon. The variations within fly ash carbon itself could not be eliminated. In addition, the original sample as well as the diluted samples was organic carbon LOI analyzed. The subsequent calibration function when applied to the CA200 readings of the calibration mixtures gave percent carbon values that were within 0.6 % carbon. This value reflected the data point scatter (worse case) relative to a smooth continuous fitted curve.

The 30 utility samples were also organic carbon LOI analyzed and compared to the CA200 reading of the same ground samples. In about a dozen cases the organic LOI analysis disagreed both higher and lower with the CA200 percent carbon indication. The worst cases being at high carbon indications. This group which showed the greater differences with the organic carbon LOI analysis were mineral analyzed. When the same samples were read with the CA200 without correcting for the zeros the correlation was improved. This was an unexpected result, however, the implication was that burning the carbon out of the zero material was in some cases creating a shift, either positive or negative with respect to the organic LOI analysis. Our tests with fly ash carbon dilution indicated that the CA200 does detect carbon and the observed shift had to be in the adsorption of light energy and the subsequent production of a sound pressure wave. There is no way to predict when this type of shift is going to occur. We would suggest that the mineral or carbon mineral reactions are responsible for the affect and when it occurs it also effects the LOI analysis. An independent highly selective analysis for elemental carbon is unfortunately not available.

Even though LOI has errors due to a variety of decompositions and carbon metal oxide reactions, the industry has become familiar with its idiosyncrasies. The calibration process was changed to adjust the original calibration curve slope to fit the organic LOI value for the sample. In addition the original calibration curve was adjusted to have the same zero value (intercept). With the calibration curve adjusted to the organic carbon LOI slope and intercept the other values agreed within 1.0% carbon of the organic carbon LOI values. This technique effectively removes the base composition variations and the two measurement methods essentially agree as long as the fly ash is from the same coal source. A before and after LOI calibration curve is shown below:

**Sample CA5, Before and After Comparison of Standard and LOI
Corrected Calibration 4/15/98**



The calibration is done when a new source of fly ash is to be measured. The organic carbon LOI or LOI value is typed in when the sample and zero are read by the CA200. The curve fitting and zeroing are done by the software. All subsequent samples will then be read by the instrument relative to the LOI and need not be changed unless one changes coals or wishes to update the calibration. The measurement process is easily done on the bench top, within the plant, within 5 minutes if sample grinding is required. Samples can be taken from different parts of the ash storage area and quickly compared to get a feel for this sort of variability as well as conveniently tracking the day to day variation in the burner performance.

We are also working on relating carbon in fly ash liquid contact surface area to our CA200 photoacoustic signal by using mercury intrusion porosimetry. We hope to report on these results at a later date.